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The Thermal and Electrical Behavior of Polyimide Films

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ABSTRACT

A series of diamines was polycondensed with pyromellitic dianhydride and 3,3',4,4'-benzophenone tetracarboxylic dianhydride using a two-step method to get thin, strong, and flexible polyimide films which were characterized by the variations of the substitution at the carbon atom connecting the two phenyl rings of the diamine moiety. The poly(amic acid) prepolymers obtained in the first step had inherent viscosities ranging from 0.9 to 1.60 and they were converted to polyimides by thermal cyclodehydration. All the polycondensation reactions were carried out in dimethyl acetamide under identical conditions. Thus 16 different polyimide polymers were synthesized and characterized by elemental analysis, IR spectroscopy, and viscosity measurements. Some other important properties, such as solubility, thermal stability, and electrical behavior, were also investigated. The alkyl chain length affected the flexibility of the resultant polyimide films greatly and the electrical properties slightly; however, thermal stability was decreased subsequently.

INTRODUCTION

The major impetus for the growth and interest in thermostable polymers is attributable to their extensive applications by the aeronautics and aerospace industries. The dire need for new high-temperature materials for supersonic and manned space flights has further enhanced the necessity for rigid research in such polymeric substances. In attempts to prepare polymers of high stability (in the thermal sense), polymer chemists have centered their attention on three major approaches [1]:

1. The synthesis of linear, single-stranded polymers based upon substituted benzonid systems, i.e., linear polyamides and polyesters.
2. The preparation of ribbonlike polymers where the system is tied together with double bonds.
3. The synthesis of ladder polymers in which aliphatic or aromatic or heterocyclic structures are joined together in an undisturbed sequence or cycle.

At short exposure at high temperature, polymers of Groups 1 and 2 exhibit some good physical properties. However, after longer exposure, the properties are severely deteriorated. On the other hand, polymers of Group 3 exhibit excellent property retention after comparable periods of time at high temperature.

The first synthesis of polyimides was obtained as early as the beginning of the 20th century [2]. However, polyimides began to be extensively prepared, studied, and utilized only in the last 20 years after DuPont established a two-step method for their synthesis. DuPont started the experimental production of polyimide films in 1962, and they now manufacture two types of films under the common trade name of Kapton. H-film is made of pure polyimide and HF-film is made of polyimide coated with polytetrafluoroethylene on one or both sides. In addition to Kapton, DuPont manufactures Vespel, Pyralin, and NR-150. Other companies manufacturing polyimide prepolymers or polyimide end polymers are Monsanto (Skyband, foamable powder), Ciba-Geigy Corp. (P13N, CR346, CR400), Rhodia Inc. (Kinel, Kermid, Nolimid adhesive), and Upjohn Co. (Polimide 2000).

As a class, polyimide polymers possess outstanding combinations of properties and retain those properties from room temperature to 500°C for a considerable period of time. Polyimides can be obtained as a thin, tough, strong film which shows good resistance to radiation, abrasion, electrical stress, and remain unaffected by many chemicals. The use of polyimides in insulating varnishes and impregnated slot linears may permit increasing the horsepower of a motor by as much as 100% with no increase in the physical size of the unit. Conversely, a motor with a given horsepower could be made much smaller. A commercial method for polyimide synthesis involves the reaction of dianhydride with diamine in a polar solvent to give poly(amic acid) which is then thermally cyclized to a high molecular weight polyimide. Detailed investigations [3] of polyimides include various methods of preparation and fabrication [4], their preparation from hydrazine [5], their reaction with hydrazine [6], their oxidative cross-linking in films [7], the effect of environmental variables on the degradation of particular polyimides [8], structure-property relationship [9-11], and properties of model compounds [12].

The condensation reactions of aliphatic aldehydes with aniline under controlled conditions give several diamines in the purest state [13]. The triphenylmethane-type diamines obtained from various aromatic aldehydes and aniline have been utilized for making polyimide films [14, 15]. The aim of the present investigation was to synthesize a series of novel polymers from various diamines and to examine the effect of increasing aliphatic chain length, at the carbon atom joining two phenyl rings, on the resulting polymer properties. The diamines used were

1. Bis(p-aminophenyl)1,1-ethane (BAPE)
2. Bis(p-aminophenyl)1,1-isobutane (BAPISO)
3. Bis(p-aminophenyl)1,1-butene (BAPBE)
4. Bis(p-aminophenyl)1,1-butane (BAPBA)
5. Bis(p-aminophenyl)1,1-hexane (BAPHEX)
6. Bis(p-aminophenyl(1,1-heptane (BAPHEPT)
7. Bis(p-aminophenyl)1,1-nonane (BAPNOE)

and the dianhydrides used were

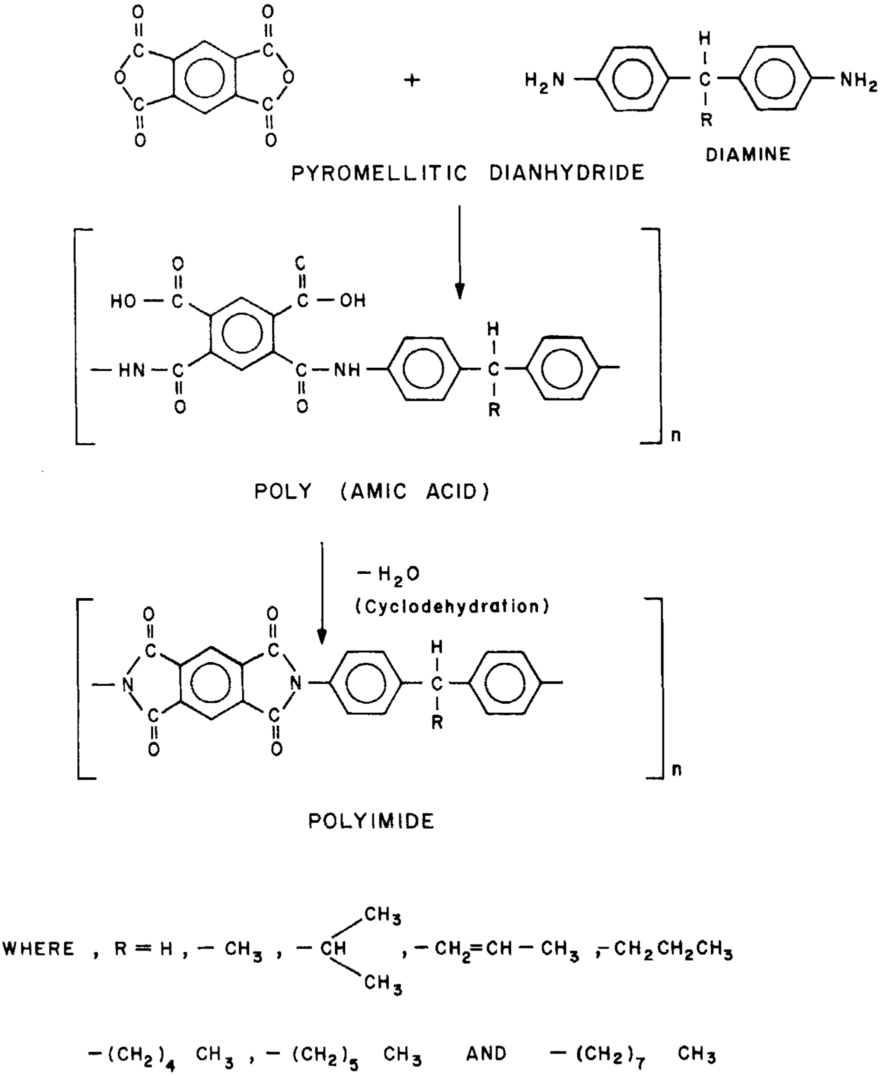
a. Pyromellitic dianhydride (PMDA)
b. 3,3',4,4'-Benzophenone tetracarboxylic dianhydride (BPTD)

In addition to these diamines, a commerically available diamine, 4,4'-diaminodiphenyl methane (4MDA), has been reacted with PMDA and BPTD to get polyimide films as end products for comparative studies. The reaction that takes place between diamine and dianhydride is given in Figs. 1 and 2.

EXPERIMENTAL

Purification of Chemicals Used

Pyromellitic dianhydride (PMDA) (Koch-Light Laboratories, England) was purified by sublimation under reduced pressure of 0.25 to 1 torr and at 230 to 250°C. BPTD was procured from Gulf Oil Co., Houston, and was also purified by sublimation at 260°C/1 torr. They were vacuum dried for 1 hr just before use. Dimethyl acetamide (DMAC) (Merck, West Germany) was kept over P_2O for 48 hr and was distilled twice. The diamines were prepared by following the method described by Ghatge and Khune [13]. Methylene dianiline (4MDA) was obtained from Bayer, A.G., West Germany, and purified



WHERE, R = H, $-CH_3$, $-CH(CH_3)_2$, $-CH_2{=}CH-CH_3$, $-CH_2CH_2CH_3$

$-(CH_2)_4CH_3$, $-(CH_2)_5CH_3$ AND $-(CH_2)_7CH_3$

FIG. 1. Synthesis of polyimides from PMDA.

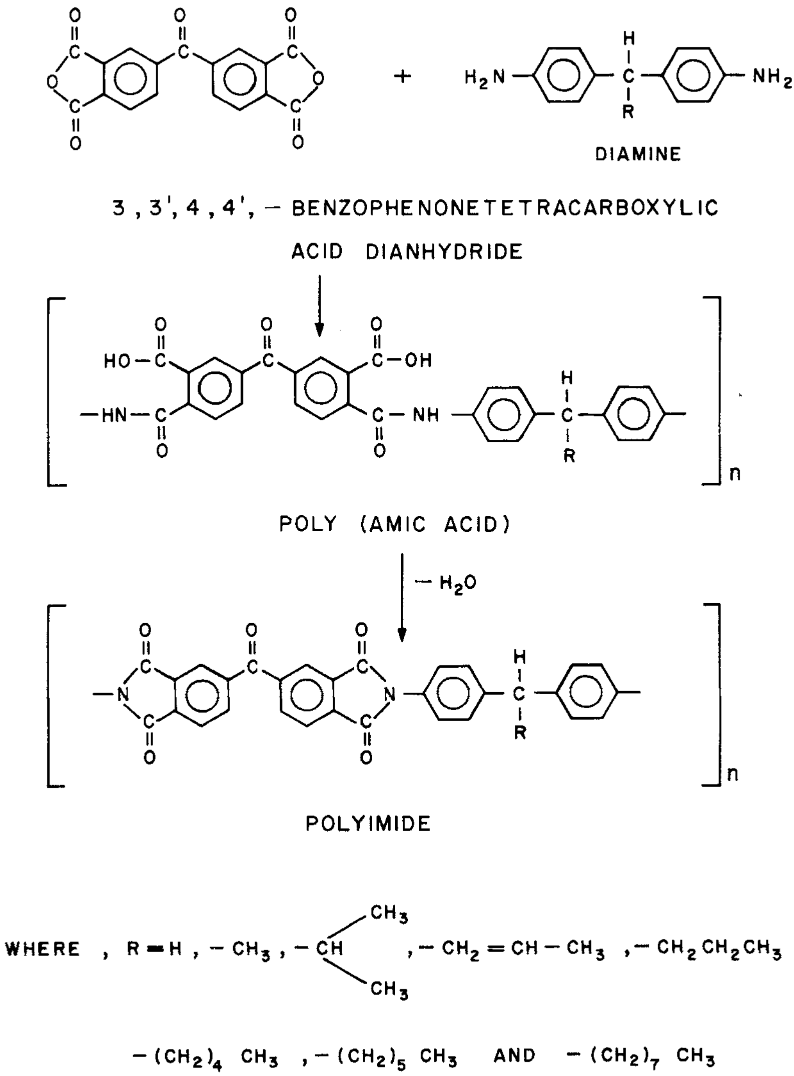


FIG. 2. Synthesis of polyimides from BPTD.

by vacuum distillation followed by recrystallization from alcohol. All diamines were perfectly vacuum dried at 40°C prior to use.

Analysis of Polyimides

The infrared spectra of the polyimide films were directly recorded on a Perkin-Elmer E-137 spectrophotometer. Thermogravimetric analysis (TGA), differential thermal analysis (DTA), and derivative thermogravimetry (DTG) were simultaneously run on a Mom Budapest derivatograph OD102 as described by Paulik et al. [16] in air at a heating rate of 9°C/min. Electrical properties were determined at room temperature. Inherent viscosity measurements were made at a 0.5% solution concentration in DMAC of poly(amic acid) and of polyimides in H_2SO_4 at 30°C. Before determining their properties, all the polyimide film samples were conditioned at 100°C under reduced pressure of 10^{-3} torr to remove the entrapped solvent and adsorbed moisture, if any.

Polymer Synthesis

Poly(amic Acid)

A 250-mL flask fitted with a magnetic stirrer, a nitrogen inlet, a drying tube, and a stopper was carefully flamed to remove traces of moisture on the walls and was allowed to cool under a stream of dry nitrogen. To this flask was added 0.01 mole of diamine followed by dry DMAC (40 to 60 mL) and the solution was well stirred. The temperature of the flask was maintained at 0°C with the help of an ice-salt mixture. Pyromellitic dianhydride (0.01 mole) or BPTD (0.01 mole) was added to the flask through a second dried powder funnel over a period of 15 min. Residual dianhydride was washed in with 5 mL of dry DMAC. The funnel was replaced by a stopper. The mixture was stirred for 1 hr, during which time the temperature of the flask was raised to 15°C. The viscous poly(amic acid) solution obtained was used for film preparation.

Poly(amic Acid) Films

In each case the poly(amic acid) solution was spread onto a dry glass plate to form a uniform layer. The solvent was removed in the inert atmosphere below 70°C. The resultant film was partly dry (about 70 to 74% solid content). The plate was cooled to room temperature, and the film was peeled off gently.

Polyimides: Conversion from Poly(amic Acid)

The above poly(amic acid) films were dried for 30 min at room temperature under vacuum and the temperature was then slowly raised. The heating schedule of Table 1 was adopted to obtain strong films.

TABLE 1

Temperature (°C)	Time	Pressure (torr)
50	1.0 hr	3
100	0.5 hr	3
125	1.0 hr	3
150	0.5 hr	3.5
200	0.5 hr	4.0
250	0.5 hr	4.5
300	10 min	5.0

RESULTS AND DISCUSSION

An investigation on the synthesis and controlled polymerization of diamines with dianhydrides has been initiated as a part of the continuing study of structure-property relationships in polyimide polymers [14, 15]. The diamine monomers were prepared in excellent yield, starting from the condensations of aniline hydrochloride and aliphatic aldehydes. These reactions were carried out in nitrobenzene solvent (165 to 170°C for 4 hr, $ZnCl_2$ catalyst), and the solvent along with excess aniline was steam distilled off after making the reaction mixture alkaline with excess sodium hydroxide. The diamine monomers thus obtained were purified either by distillation under reduced pressure or by recrystallization (from ethyl alcohol or carbon tetrachloride).

A well-established fact in polyimide preparation is that addition of dianhydride to the diamine solution gives a polyimide of high molecular weight than does the reverse addition. A stoichiometric amount of dianhydride was added to the yellowish to red solutions of diamine in DMAC to get the respective poly(amic acids). Upon completion of the addition of the dianhydride, the polymer solution was stirred for 1 hr at 0 to 15°C before the poly(amic acid) films were cast. The reactions were carried out under perfectly dry conditions since the presence of moisture, even in trace amount, causes hydrolysis of poly(amic acid) and hinders cyclodehydration. Although the polycondensation proceeds readily at 0 to 15°C in DMAC, only 0.9 to 1.6 dL/g inherent viscosity poly(amic acids) were obtained. The use of higher temperatures did not induce higher viscosities.

Poly(amic acid) films were obtained by casting the polymer solutions onto dry glass plates and removing the solvent below 70°C in an inert atmosphere. To obtain the poly(amic acids) in a powdered form [since the IR spectrum of poly(amic acid) film failed to show the N–H band due to thickness and solvent content (30%)], the prepolymer was precipitated by distilled water, filtered, washed with acetone, and dried. The IR spectra of powdered poly(amic acids) were recorded in Nujol mull. As an illustration, a spectrum of poly(amic acid) obtained in turn from BAPBA and PMDA is given in Fig. 3 which shows a characteristic N–H adsorption at 3250-3450 cm^{-1}. Infrared spectra of all the polyimide films were recorded directly without the use of any medium. Each spectrum exhibited bands at 1780, 1730, 1380, and 730 cm^{-1}, which are characteristic of the imide unit (for an illustration, see Fig. 4). In the case of polyimides obtained from the diamine BPTD reaction, a peak of comparable intensity is observed at about 1670 cm^{-1} which can be assigned to the benzophenonic carbonyl group. The IR spectrum of BPTD also shows adsorption at this frequency. All the polyimide polymers gave elemental analysis in excellent agreement with the theory (see Table 2). However, polymers prepared from 4MDA, BAPE, PMDA, and BPTO showed analytical results less than theoretical. This may be due to their tremendous resistance to ionization, even at high temperatures.

Polyimide films were insoluble in chloroform, ethanol, benzene, toluene, hexane, chlorobenzene, o-dichlorobenzene, and nitrobenzene. Viscosity measurements of polyimide polymers were made in H_2SO_4. The viscosities decreased with an increase in chain length at the hinge "CH_2." Some other properties of polyimide polymers are described in Table 3.

It is important to note that the information obtained via thermal weight loss studies provide valuable data concerning the stability of polymers at high temperatures (Table 4). In all cases the information from weight loss data is supplemented by examination of the differential thermal analysis (DTA) behavior of the polymer system. In spite of such limitations as the lack of information regarding physical property changes, these methods serve as sources of data illustrative of the difference between polymers of widely varying structure.

The weight losses in polymers when heated under dynamic conditions at a heating rate of 9°C/min are shown in Figs. 5 to 20. T_0 is the initial temperature up to which a polymer retains its principal chemical structure. After the removal of easily volatile materials (losses up to 2 ± 1.5%), the weight of the polymer is stabilized to T_0 and suddenly starts dropping due to an acceleration of the polymer chain breaking-down process. Koton et al. [17, 18] have observed that the structure of the diamine monomer affects the thermal stability. Various polyimides having different diamine components and a similar dianhydride component have been arranged in Table 5 in the order of thermal stability depending on the value of T_0 obtained.

It seems from the Table 5 data that the thermal stability of a polymer is higher if symmetry exists in the hinge group. Asymmetric hinge groups tends to reduce the stability of polymers. By and large, a similar trend was observed in the case of polymers synthesized in the present investigation. Polymers I and IX, having symmetry in the diamine moiety, showed values of T_0 450 and 440°C, respectively, while the rest of the polyimide polymers having

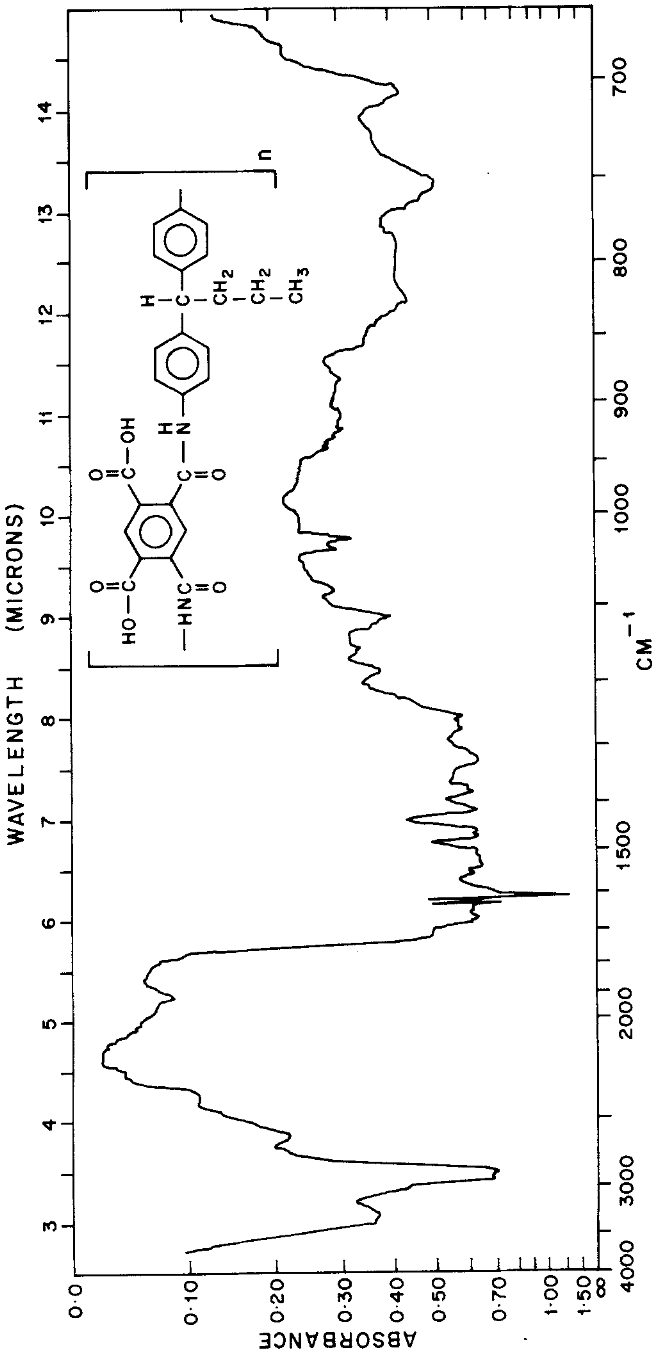


FIG. 3. IR spectrum of poly(amic acid) from BAPBA and PMDA reaction.

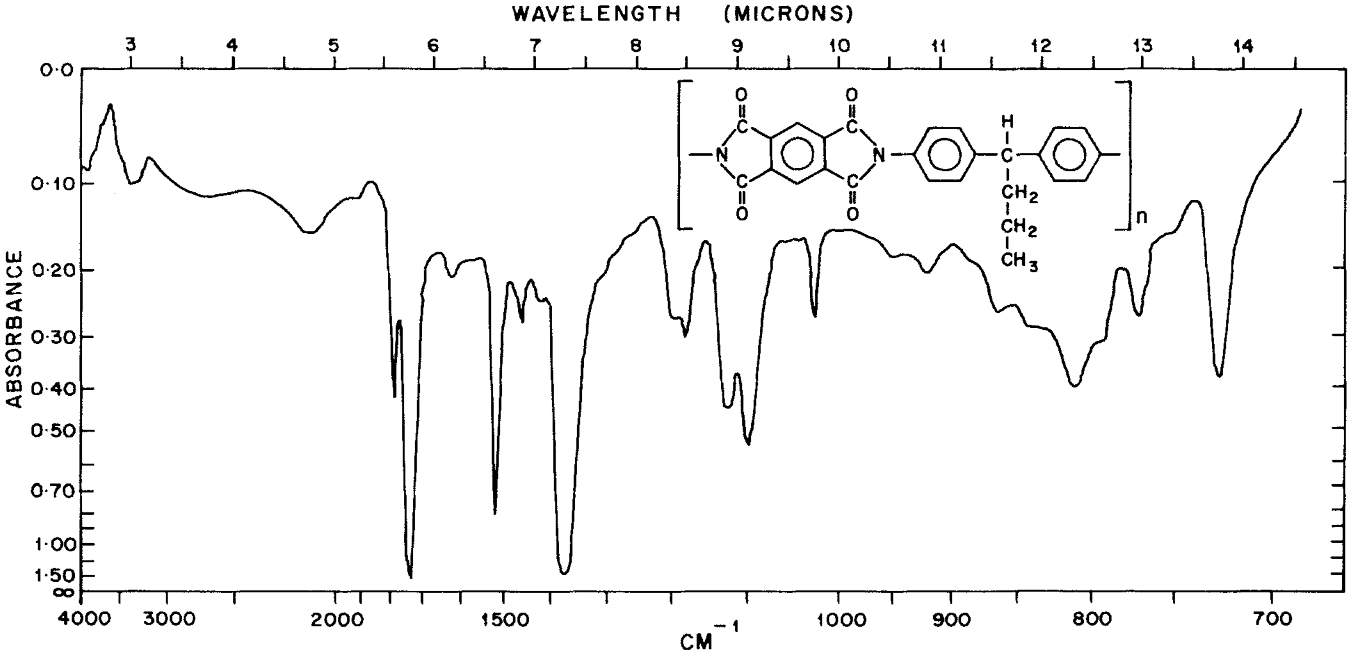


FIG. 4. IR spectrum of polyimide from BAPBA and PMDA reaction.

TABLE 2. Reactants, Reaction Conditions, and Properties of Prepolymers and End Polymers Obtained

No.	Reactants quantities (g)	Quantity of DMAC (mL)	Inherent viscosities (dL/g)		Elemental analysis of polyimide end polymer			
			Prepolymer in DMAC	End polymer in H_2SO_4		C	H	N
I	MDA + PMDA (1.98) (2.18)	41.60	1.6037	1.2004	Calcd. Found	72.63 70.23	3.01 2.10	7.36 5.12
II	BAPE + PMDA (2.12) (2.18)	43.00	1.3562	1.0136	Calcd. Found	73.09 71.82	3.55 2.26	7.11 6.36
III	BAPISO + PMDA (2.40) (2.18)	45.80	1.2046	0.9321	Calcd. Found	73.93 73.08	4.26 4.12	6.61 6.50
IV	BAPBE + PMDA (2.38) (2.18)	45.60	1.2138	0.9265	Calcd. Found	74.27 74.00	3.81 2.54	6.66 6.49
V	BAPBA + PMDA (2.40) (2.18)	45.80	1.1012	0.9272	Calcd. Found	73.93 73.75	4.26 4.10	6.61 6.42
VI	BAPHEX + PMDA (2.68) (2.18)	48.60	0.9546	0.8355	Calcd. Found	76.66 76.28	4.88 4.60	6.22 6.10
VII	BAPHEPT + PMDA (2.84) (2.18)	50.20	0.9409	0.8310	Calcd. Found	75.00 74.66	5.16 5.04	6.03 5.97
VIII	BAPNOE + PMDA (3.10) (2.18)	52.80	0.9382	0.8287	Calcd. Found	75.61 65.43	5.69 5.57	5.69 5.60
IX	MDA + BPTD (1.98) (3.22)	52.00	1.5061	1.1768	Calcd. Found	74.36 72.02	3.30 1.17	5.78 4.15
X	BAPE + BPTD (2.12) (3.22)	53.40	1.3006	1.0024	Calcd. Found	74.69 73.26	3.61 2.40	5.62 4.17

(continued)

TABLE 2 (continued)

No.	Reactants quantities (g)	Quantity of DMAC (mL)	Inherent viscosities (dL/g)		Elemental analysis of polyimide end polymer			
			Prepolymer in DMAC	End polymer in H_2SO_4		C	H	N
XI	BAPISO + BPTD (2.40) (3.22)	56.20	1.2245	0.9206	Calcd.	75.47	4.18	5.32
					Found	75.13	4.00	5.10
XII	BAPBE + BPTD (2.38) (3.22)	56.00	1.1837	0.0119	Calcd.	75.55	3.81	5.34
					Found	75.48	3.73	5.23
XIII	BAPBA + BPTD (2.40) (3.22)	56.20	1.1286	0.9075	Calcd.	75.47	4.18	5.32
					Found	75.14	3.98	5.06
XIV	BAPHEX + BPTD (2.68) (3.22)	59.00	1.0063	0.8310	Calcd.	75.81	4.68	5.05
					Found	75.77	4.52	4.90
XV	BAPHEPT + BPTD (2.84) (3.22)	60.60	0.9612	0.8291	Calcd.	76.05	4.92	4.92
					Found	75.83	4.80	4.73
XVI	BAPNOE + BPTD (3.10) (3.22)	63.20	0.9298	0.8206	Calcd.	76.51	5.36	4.69
					Found	76.40	5.08	4.38

TABLE 3. General Properties of Polyimide Polymers (Film)

Polyimide	Effect of organic solvent	Clarity	Effect of sunlight	Effect of		Burning possibility
				Strong acid	Alkali	
I	None	Opaque	None	Attacked	Yes	None
II	None	Opaque	None	Attacked	Yes	None
III	None	Opaque	None	Attached	Yes	None
IV	None	Opaque	None	Attacked	Yes	None
V	None	Opaque	None	Attacked	Yes	None
VI	None	Opaque	None	Attacked	Yes	None
VII	None	Opaque	None	Attacked	Yes	None
VIII	None	Opaque	None	Attacked	Yes	None
IX	None	Opaque	None	Attacked	Yes	None
X	None	Opaque	None	Attacked	Yes	None
XI	None	Opaque	None	Attacked	Yes	None
XII	None	Opaque	None	Attached	Yes	None
XIII	None	Opaque	None	Attacked	Yes	None
XIV	None	Opaque	None	Attacked	Yes	None
XV	None	Opaque	None	Attacked	Yes	None
XVI	None	Opaque	None	Attacked	Yes	None

TABLE 4. Weight Loss at Different Temperatures for Polyimide (%)

	Temperature (°C)								
Polyimide	100	200	300	400	500	600	700	800	900
I	0.00	0.1	1.50	2.10	4.75	25.10	36.00	49.00	62.00
II	0.75	1.00	2.00	2.25	9.50	31.10	41.00	50.1	66.03
III	0.75	1.15	2.20	3.15	10.15	30.00	42.25	56.00	64.50
IV	1.00	1.25	2.30	3.75	10.40	30.15	43.00	57.00	66.00
V	1.00	1.50	2.75	4.00	10.50	31.00	43.00	58.50	65.60
VI	1.25	1.60	3.00	5.25	11.15	32.00	95.00	59.00	67.50
VII	1.25	1.65	3.00	6.00	11.45	32.25	45.50	59.00	67.25
VIII	1.35	1.75	4.00	6.75	12.25	33.15	47.00	60.15	68.13
IX	0.25	1.00	2.00	2.k5	4.85	26.15	37.00	49.85	63.15
X	0.85	1.15	2.10	2.75	10.000	31.00	42.15	50.25	66.75
XI	1.00	1.25	2.30	3.45	11.00	32.10	43.00	52.00	65.25
XII	1.10	1.25	2.40	4.00	11.75	33.00	44.20	57.00	66.50
XIII	1.05	1.20	2.50	4.50	10.20	33.15	44.25	57.25	66.75
XIV	1.20	1.65	3.15	5.50	11.35	33.75	46.00	59.50	68.50
XV	1.40	1.80	3.25	6.80	11.65	34.15	47.50	61.10	68.50
XVI	1.40	1.80	3.25	6.95	12.80	34.25	48.00	61.00	69.00

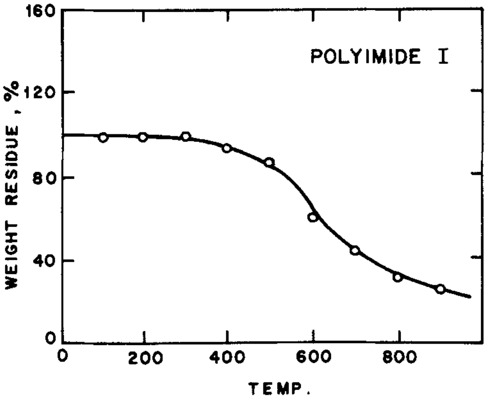


FIG. 5

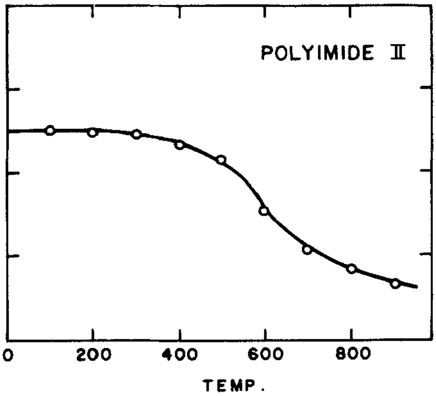


FIG. 6

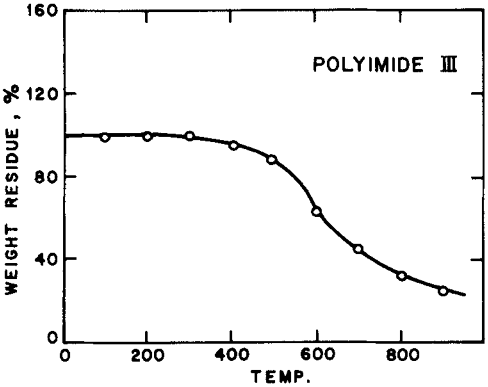


FIG. 7

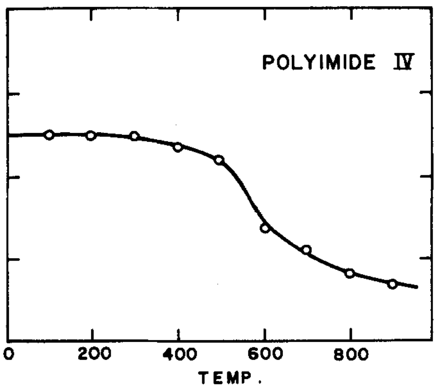


FIG. 8

FIGS. 5-8. TGA curves in air at 9° C/min.

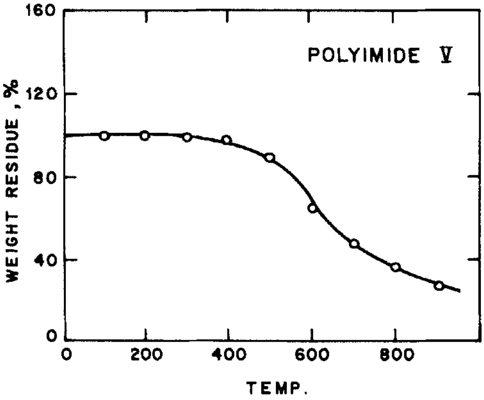


FIG. 9

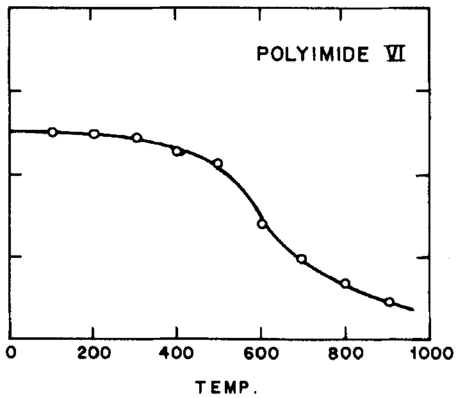


FIG. 10

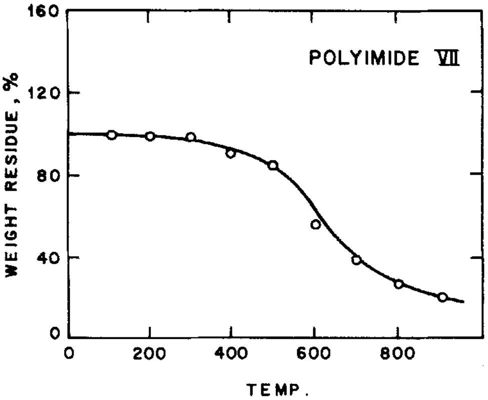


FIG. 11

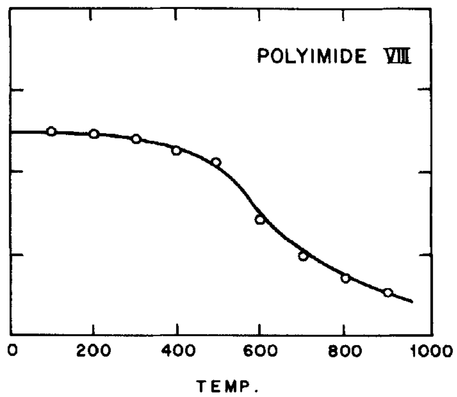


FIG. 12

FIGS. 9-12. TGA curves in air at 9° C/min.

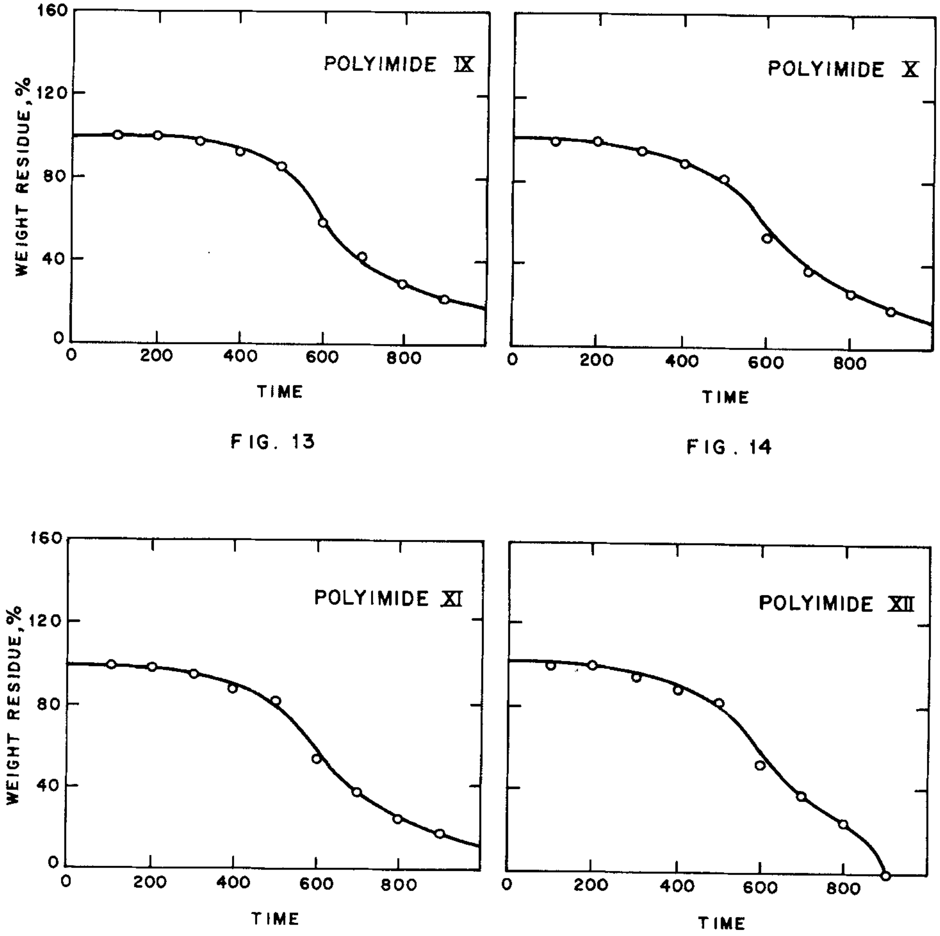


FIG. 13

FIG. 14

FIG. 15

FIG. 16

FIGS. 13-16. TGA curves in air at 9°C/min.

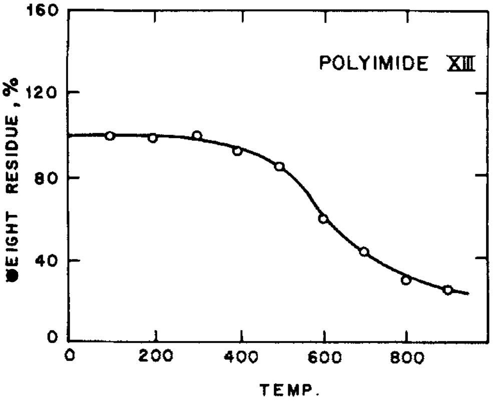


FIG. 17

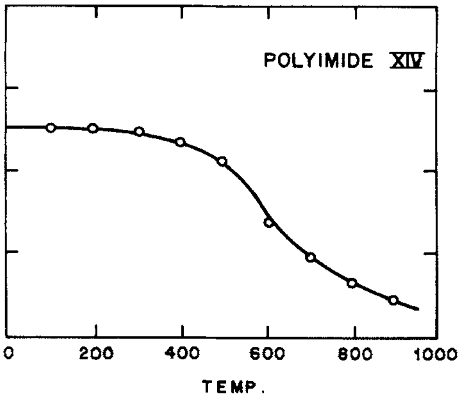


FIG. 18

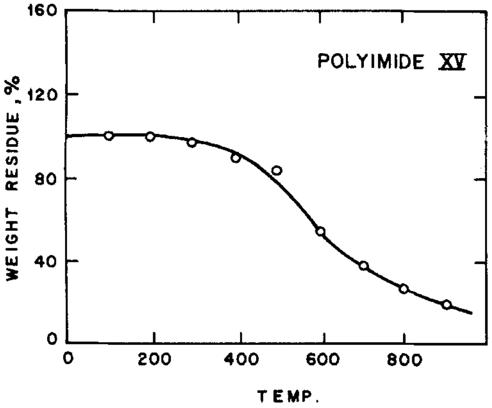


FIG. 19

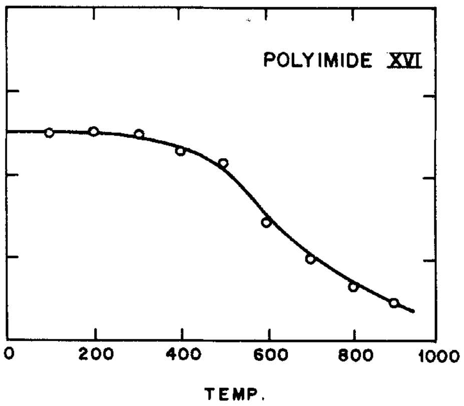
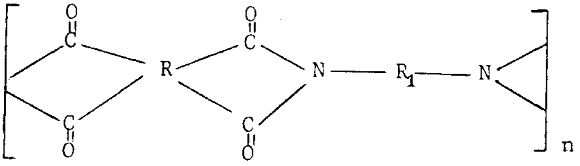
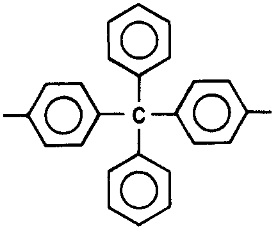
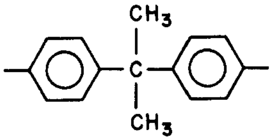
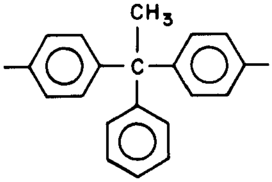


FIG. 20

FIGS. 17-20. TGA curves in air at 9° C/min.

TABLE 5

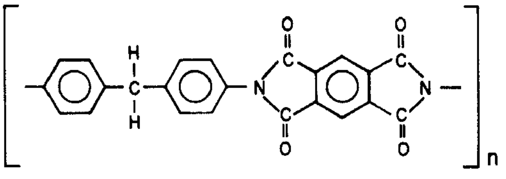
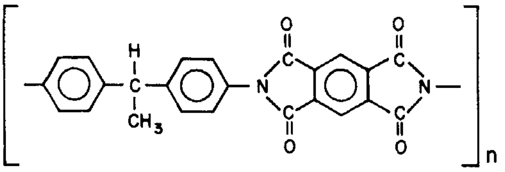
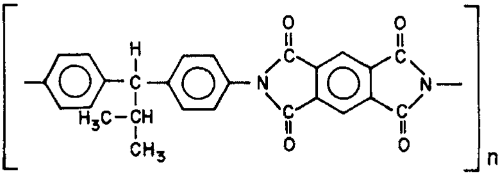
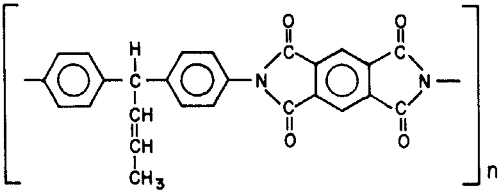
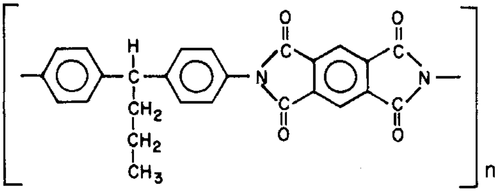
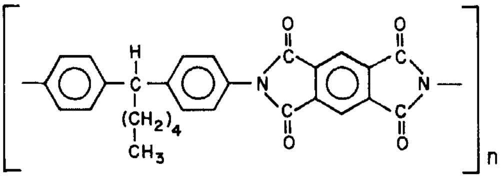


Repeat unit (R_1)	T_0 (°C)
	400
	360
	350

an asymmetric structure are less stable. As the aliphatic chain length at the hinge carbon atom increases, the value of T_0, and hence the stability of the polymers, decreases. This is true in both types of imide polymers from PMDA and BPTD.

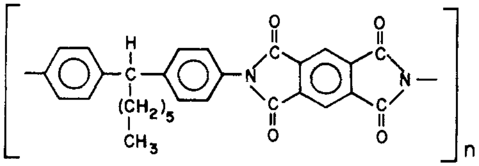
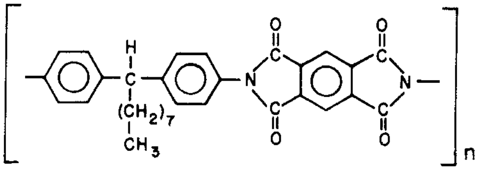
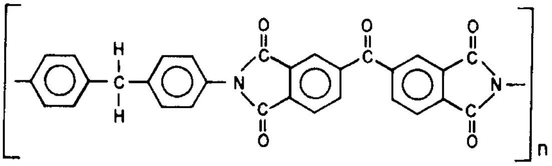
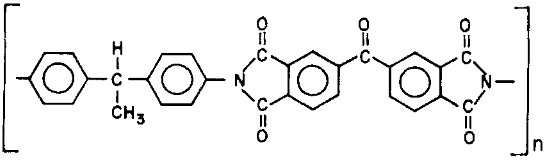
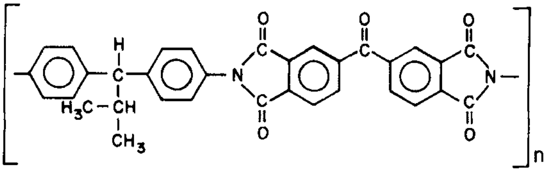
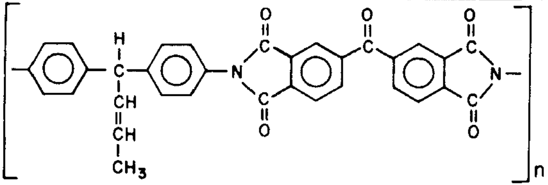
The temperature for 10% weight loss, T_{10}, characterizes the moment of thermal decomposition of polyimides when processes of weight loss due to elimination of highly volatile products are basically complete.

TABLE 6. Temperature (°C) for 0, 10, and Maximum Weight Loss

NO.	POLYIMIDE	T_0	T_{10}	$T_{MAX.}$	LOSS AT 900°C
I		450	530	650	63·15
II		390	450	635	66·75
III		385	425	625	65·25
IV		380	425	615	66·50
V		375	415	610	66·75
VI		360	405	600	68·50

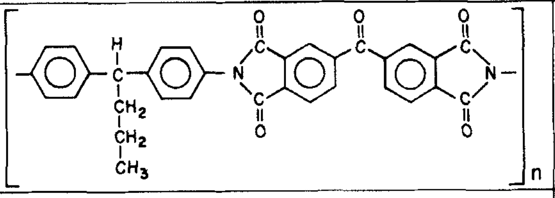
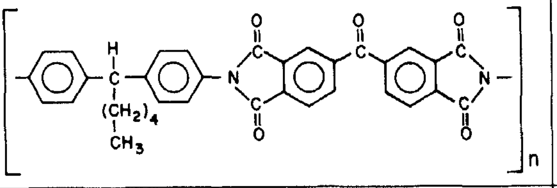
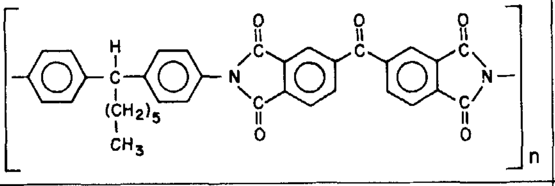
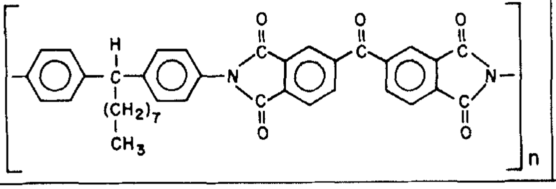
(continued)

TABLE 6 (continued)

NO	POLYIMIDE	T_0	T_{10}	$T_{MAX.}$	LOSS AT 900°C
VII		350	400	595	68·50
VIII		340	400	590	69·00
IX		440	515	645	62·00
X		385	440	620	66·03
XI		380	415	600	64·50
XII		375	415	585	66·00

(continued)

TABLE 6 (continued)

NO	POLYIMIDE	T_0	T_{10}	$T_{MAX.}$	LOSS AT 900°C
XIII		370	415	585	65·60
XIV		355	400	585	67·50
XV		340	395	580	67·25
XVI		340	390	580	68·13

Usually, this is supposed to be the initial stage of breakdown of the main imide chain. Thus more thermally stable polymers possess high T_{10} value. T_{10} values can be meaningfully correlated to the present class of polymers. It is seen from Tables 4 and 6 that the polymers containing symmetric hinges (–CH_2–) have T_{10} values of 530 and 515°C whereas the T_{10} values for other polymers are far less than 450°C.

The temperature for the maximum rate of decomposition (T_{max}) of polymers either from PMDA or BPTD varies over a narrow range of temperature (620 ± 30°C for polyimides from PMDA and 620 ± 20°C for polyimides from BPTD). On reaching T_{max}, the weight loss of polymers is about 55 to 60%. This indicates the stage of decomposition of the imide structure of the polymer chain.

TABLE 7. Electrical Behavior of Polyimide Films

Polyimide	Volume resistivity (ρ)	Dielectric constant (ϵ)		Dissipation factor (tan δ)	
		1 kHz	10 kHz	1 kHz	10 kHz
I	2.16×10^{16}	3.20	3.14	0.0022	0.0131
II	2.07×10^{16}	2.87	2.80	0.0020	0.0128
III	2.00×10^{16}	2.78	2.76	0.0021	0.0114
IV	2.12×10^{16}	3.10	3.02	0.0024	0.0110
V	1.96×10^{16}	2.91	2.83	0.0021	0.0109
VI	2.14×10^{16}	3.00	2.96	0.0019	0.0107
VII	2.00×10^{16}	2.92	2.85	0.0020	0.0110
VIII	$x.00 \times 10^{16}$	3.01	2.97	0.0020	0.0112
IX	1.90×10^{16}	3.00	2.95	0.0020	0.0110
X	2.00×10^{16}	2.86	2.83	0.0025	0.0125
XI	1.89×10^{16}	3.26	3.06	0.0021	0.0110
XII	1.95×10^{16}	2.99	2.93	0.0022	0.0123
XIII	2.00×10^{16}	3.03	2.98	0.0023	0.0116
XIV	2.15×10^{16}	2.85	2.80	0.0018	0.0121
XV	2.19×10^{16}	3.01	3.00	0.0016	0.0103
XVI	1.93×10^{16}	2.94	2.90	0.0015	0.0101

ELECTRICAL PROPERTIES

Electrical properties, such as volume resistivity (ρ), dielectric constant (ϵ), and loss factor (tan δ), of all the polyimide films are illustrated in Table 7. The volume resistivity—a measure of the ratio of the potential gradient parallel to the current in the film to the current density—is found to be of the order 10^{16} and numerically varies from 1.89 to 2.16. The dielectric constant, a ratio of the capacitance of a parallel plate condenser measured with and without the polyimide film placed between the plates, ranges from 2.28 to 3.26. The polarization of the dielectric makes this change. Time dependency of applied field leads to time-dependent polarization. However, the atoms in the dielectric polyimide film resist moving, causing a delay between changes in the applied field and a change in polarization. This delay is nothing but a phase difference or loss angle δ. Sin δ is termed a power factor and tan δ a dissipation factor. In the present class of polymers (films), both the dielectric constant and the dissipation factor vary slightly from one polymer to another since there is no prominent polar group present in any polyimide. These values are quite comparable to those polyimide films commercially available. However, cellulose acetate and poly(vinyl alcohol) films have better values than polyimides in general.

CONCLUSION

Polyimide films of excellent strength can be prepared from poly(amic acid) which, in turn, is obtained by the reaction of a diamine with a dianhydride in a polar solvent such as DMAC. The symmetry in the repeat unit of the polymer affects the thermal stability of the polyimide polymers. The more the symmetry, the higher the stability. In the absence of polar substituent on the repeating unit, symmetry contributes less to the electrical properties of the polyimide films. A diamine-PMDA reaction produced a better quality of polyimide film than a diamine-BPTD reaction. The polyimide films thus obtained have excellent resistance to organic solvents; however, they are affected by concentrated sulfuric acid, nitric acid, hydrochloric acid, and alkalies.

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